IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Wu et al.

Application No.: 10/820,525

Filed: April 7, 2004

Title: METHODS FOR PRODUCING LOW-K CDO FILMS WITH LOW RESIDUAL STRESS

Attorney Docket No.: NOVLP091/NVLS-2889

Examiner: Maldonado, Julio J.

Group: 2823

CERTIFICATE OF EFS-WEB TRANSMISSION
I hereby certify that this correspondence is being transmitted electronically through EFS-WEB to the Commissioner for Patents, P.O. Box 1450 Alexandria, VA 22313-1450 on March 4, 2008.

Signed: /Tara Hayden/
Tara Hayden

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Qingguo Wu, declare as follows:

CONCEPTION

1. I am a co-inventor of the subject matter claimed in claims 1-9, 12-33, 34 and 35 of the above-referenced patent application.

2. Prior to January 16, 2004, my co-inventors and I invented the subject matter claimed in claims 1-9, 12-33, 34 and 35 of the patent application cited above. Specifically, we conceived the invention in the United States before that date. In addition, we drafted an "Invention Priority Data and Information" document describing the invention. This document is attached as Exhibit A and we prepared it prior to January 16, 2004. The specific dates evidencing conception and other confidential information have been redacted from this document.

3. As shown, the "Invention Priority Data and Information" document (Exhibit A) describes producing low-k carbon-doped oxide (CDO) films having low stress. Regarding independent claims 1, 19 and 29, please see the "Abstract" and "Technical Contents." Of particular relevance, see "Process Optimization" on pages 7 and 8, in which forming a CDO film having < 35 MPa and a k < 3.0 is described.

4. I am informed and believe that prior to January 16, 2004, my co-inventor Haiying Fu discussed the invention via telephone conference with Joseph Bond and Jeffrey Weaver, the patent attorney and patent agent responsible for drafting the application. I am informed and believe that Jeffrey Weaver notes during this conversation, which were kept in the attorney file for this case. Two pages of these pages containing portions relevant to aspects of our invention are attached as Exhibit B. Regarding independent claim 29, see the LFRF frequency ranges on diagram on both attached pages.

DILIGENCE

Attached Exhibits C-F show the diligent pursuit of the completion of the invention from a time just prior to the effective date of the cited prior art (January 16, 2004) to the time of filing of the application (April 7, 2004). I am informed and believe that copies of these documents were located in the attorney file for this case.

1. Exhibit C is a copy of a fax coversheet dated January 12, 2004 from Joseph Bond, a patent agent involved with drafting the application, to Jeffrey Weaver, the attorney responsible for drafting the application enclosing a draft of a figure (labeled Fig. 13 in the draft and what became Figure 14 of the application) for this case (NOVLP091/NVLS-002889) for Jeffrey Weaver's review.

2. Exhibit D is a copy of an email from Joseph Bond to my co-inventor Haiying Fu enclosing a draft of the application, dated February 5, 2004.

3. Exhibit E is a copy of an email string showing my co-inventor Haiying Fu sent a revised draft to Jeffrey Weaver and Joseph Bond on February 22, 2004. The string also shows that Jeffrey Weaver and Joseph Bond modified the draft on a date after that and requested a phone conversation with my co-inventor regarding the application. Confidential information has been redacted from this document.

4. Exhibit F is a copy of a letter dated March 29, 2004 from Joseph Bond to Haiying Fu enclosing a revised draft of the application. The letter also states that changes were made to the draft based on inventor comments and encloses a declaration to be signed.

5. The patent application was filed on April 7, 2004.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. I further declare that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both (under Section 1001 of Title 18 of the United States Code), and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

3/4/2008

Qingguo Wu Date

EXHIBIT A

Return completed forms to Roland Tso M/S 280

NOVELLUS
FIRST IN PRODUCTIVITY

INVENTION PRIORITY DATA AND INFORMATION

Docket Number: 2889 Date Received:

1. Title of Invention: Methods for producing Low-k OSG film of low tensile stress
2. Description of Invention: (Please attach witnessed "Invention Disclosure" forms to this one.)
3. Invention conception date: _____
4. Date of first written description of the Invention (attach copy): _____
5. First disclosed to: _____ Date: _____
6. Date of first offer for sale of product incorporating the Invention: _____
7. Date of first or future shipment to customer (name & date): _____
8. Date of first or future publication of the Invention: _____
9. List the products which the Invention will be used in: _____

INVENTOR(S):

1. Name: Haiying Fu Employee Number: 2434
   Work Address: 11155 SW Leveton Dr, Tualatin, OR 97062
   Work Phone Number: 503.885.6625 Fax Number: 503.612-8701
   Home Address: 22580 Clark Street, West Linn, OR 97068
   Signature: _____ Date: _____ Citizenship: USA
2. Name: QINGGUO Wu Employee Number: 5810
   Work Address: 11155 SW Loveton Dr. Tualatin, OR 97062
   Work Phone Number: 503-885 6749 Fax Number: 503-612 8701

NOVLP091

Home Address: 17865 SW 113th Ave. Tualatin, OR 97062

Signature: [signature] Date: Citizenship: Canada

3. Name: Dong Niu Employee Number: 5905

Work Address: 11155 SW Leveton DR, Tualatin, OR 97062

Work Phone Number: 503-885-6459 Fax Number: 503-612-8701

Home Address: 10845 SW Royalty Pkwy, Apt L32, Tigard, OR 97024

Signature: [signature] Date: Citizenship: China

WITNESS DECLARATION (must be signed and dated by two witnesses):

I have read and examined the above, the description on the attached disclosure forms, and I understand the subject matter described therein.

1. First Witness (print): XINGYUAN TANG

Signature: [signature] Date:

2. Second Witness (print): A K BANDYOPADHYAY

Signature: [signature] Date:

INVENTION DISCLOSURE

Novellus Systems, Inc.

Title of Invention: **Methods for producing Low-k OSG Film of low tensile stress**

Date this form was completed: ·

Abstract

The present invention involves a chemical vapor deposition (CVD) reactor applying different deposition conditions, including process gases and process parameters, to form low-k films. The present invention relates to process optimization and improvement, through precursor structure optimization and process parameter optimization, to lower CDO film tensile stress to below 30Mpa or even compressive stress.

Haiying Fu Signature

Qingguo Wu Signature

Dong Niu Signature

Inventor Signature

Signature of 1st Witness / Date

Signature of 2nd Witness / Date

INVENTION DISCLOSURE

Novellus Systems, Inc.

Title of Invention: **Methods for producing Low-k OSG Film of low tensile stress**

Date this form was completed:

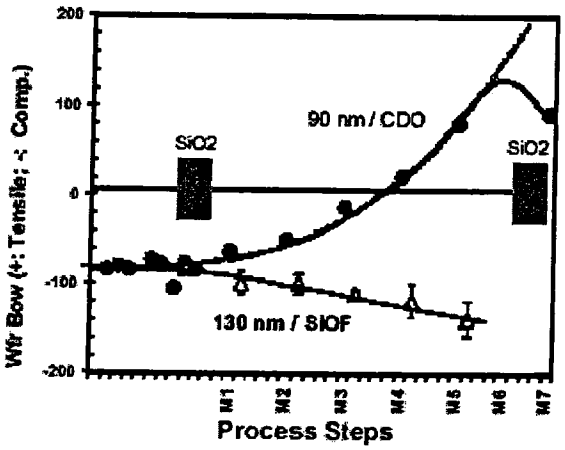

Figure 1. Wafer level stress as a function of process steps for 90 nm technology and 130 nm SiOF technology. Note that the tensile stress development in CDO stacks is offset by the compressive stress of the oxide layer at the top.

1. The tensile stress of low k film will be lowered through film densification
2. To achieve densification while maintaining low k value, precursor structure optimization is suggested.
3. To achieve densification while maintaining low k value, process conditions need to be optimized to improve crosslinking to enhance Si-CH_2-Si bonding content
4. Densified film with high Si-CH_2-Si content will have low stress and high hardness/modulus, thus better cracking resistance.

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Qingguo Wu Signature

Dong Niu Signature

Inventor Signature

Signature of 1st Witness / Date

Signature of 2nd Witness / Date

5. Percursor optimization includes more methyl groups in the precursor and preferred structures.
6. Process optimization includes process conditions to improve –CH3 incorporation in the film and to enhance crosslinking of –CH3 groups to form Si-CH2-Si matrix besides the existing Si-O-Si cage structure.

Technical Contents

The CDO film was deposited by using plasma enhanced CVD (PECVD) technology. Chemical precursor(s) with carrier gas such as CO_2 was introduced into a vacuum chamber, where RF plasma was ignited to polymerize the precursor and CDO was deposited onto a substrate. The suitable precursors include multiple siloxanes, such as tetramethylcyclotetrasiloxane (TMCTS), octamethylcyclotetrasiloxane (OMCTS), methyl-dimethylsiloxane (M-DMOS), and trimethyl-methylsiloxane (TM-MOS). Other suitable precursors include alkylsilanes, such as 4MS, TMSA, BTMSA, vinyltrimethylsilane (VTMS, SiC_5H_{12}), hexamethyldisilane (HMDS, $Si_2C_6H_{18}$). Further suitable precursors include the mixture of siloxane and alkylsilane.

The stress of CDO in an unoptimized process conditions was generally >50 Mpa with a typical value in the range between 60Mpa and 90 MPa. In this invention, we described the method on how to low the film stress through optimizing precursor and process conditions.

To reduce the film tensile stress, it is desired that the CDO film as deposited increase in its density while k value remain the same, e.g. by applying low frequency power and incorporating more carbon content and carbon associated bonding structures. Since methyl or

Haiying Fu Signature	Qingguo Wu Signature
Dong Niu Signature	Inventor Signature
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methylene group has a low polarizability, thus its increase in content in the deposited film will not have significant adverse impact on the dielectric constant, which is a crucial evaluation measure of a CDO film. The increase in carbon content incorporated in the film will increase film refractive index and density, and hence lower the tensile stress of the film.

It is also desired that the process deposition condition be optimized to enhance/promote the crosslinking of methylene group incorporated in the film to form Si-CH_2-Si structure. Without this crosslinking, the film mechanical strength will generally low due to the fact that an increase in content of methyl group CH_3 as a terminating group in the CDO film will reduce the bonding density per volume thus the bonding integrity of the film. With the crosslinking of methylene group, the Si-CH2-Si bonds are formed to strengthen the existing Si-O-Si cage structure. As a result, the mechanical strength, or the toughness, of the as deposited film will remain intact. The toughness is a measure of the resistance of a film against cracking propagation once the cracking is initiated. The crosslinking of Si-CH_3 groups will not increase the C content of the film, while Si-O-Si structure is still dominating the film composition to maintain a low dielectric constant. This ensures that the etching characteristic of the film as deposited should not be significantly altered.

The formation of Si-CH_2-Si is evident by FTIR spectra of the as deposited film. Figure 2 shows that one film with higher refractive index has bigger peak (see inset of Figure 2) assigned to bending of C-H in Si-CH_2-Si crosslinks although the k values of film a and b are the same.

Haiying Fu Signature

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Inventor Signature

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Signature of 2nd Witness / Date

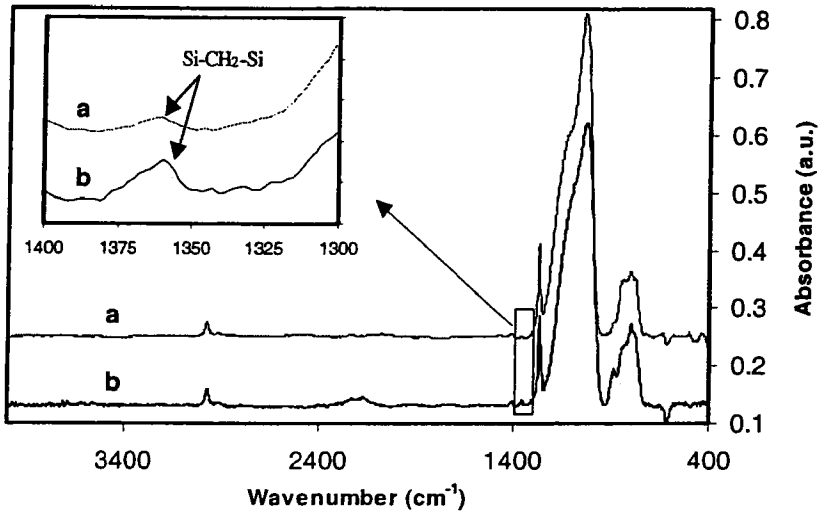

Figure 2. FTIR spectra of two CDO films: "a" and "b" with the same k = 2.94 and hardness H~2.02GPa. The difference in the Si-CH2-Si results in difference in other properties of these two films, including RI and film stress.

The formation of Si-CH_2-Si bonding structure is also evident by refractive index of the as-deposited film. Table below summarized the properties of Film "a" and Film "b". Though k value and hardness are similar for these two films, the difference in film bonding structure, especially Si-CH_2-Si bond as shown in Figure 2, results in that the two films has significantly different refractive index (RI) and in-film stresses. Film "b" of higher refractive index has a lower tensile stress than film "a". The additional bonding in film "b" also improved its cracking limit over film "a".

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Title of Invention: **Methods for producing Low-k OSG Film of low tensile stress**

Date this form was completed:

	K	H (GPa)	RI	Stress (MPa)	Cracking Limit (um) HF
Film "a"	2.94	2.02	1.3913	88.5	1.5 μm
Film "b"	2.94	2.06	1.4404	67.9	4.3um

Figure 3 shows more data to illustrate the relationship of as deposited film stress and refractive index. RI is used as a measure of volumetric content of Si-C-Si in the film. The tensile stress decreases below 50MPa as RI or content of Si-C-Si bonds increases.

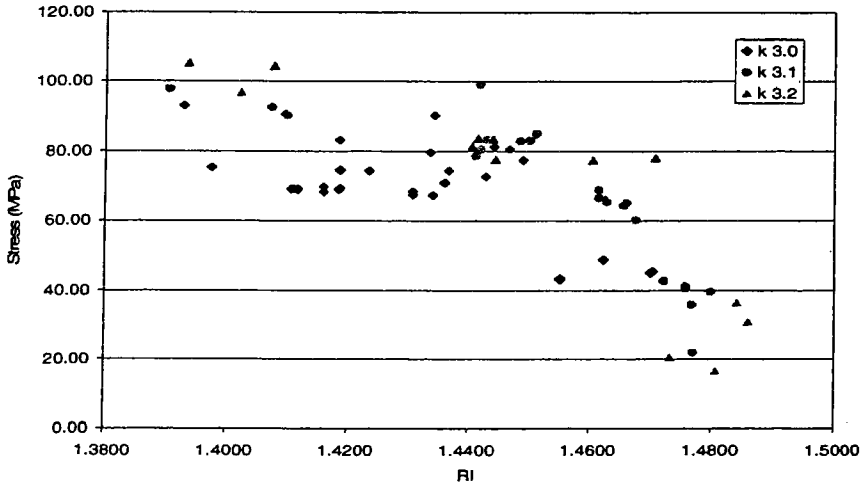

Figure 3. Stress is plotted as a function of refractive index of as deposited film for different k values. For all k values, stress decreases as refractive index increases.

To optimize the as deposited film bonding structure to lower the film tensile stress, two key areas are found to be critical.

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1. Precursor selection. Precursor structure has a significant impact on the structure of the film deposited. The precursor structure prefers containing more $-CH_3$ group as shown in the Figure 4. More –CH3 group in the precursor will enhance CH3 content incorporated in the deposited film to increase as-deposited film density thus lower the film stress. Process conditions are further optimized to promote certain bonding formation such as $Si-CH_2-Si$ to further strengthen the film mechanical property and lower film stress. .

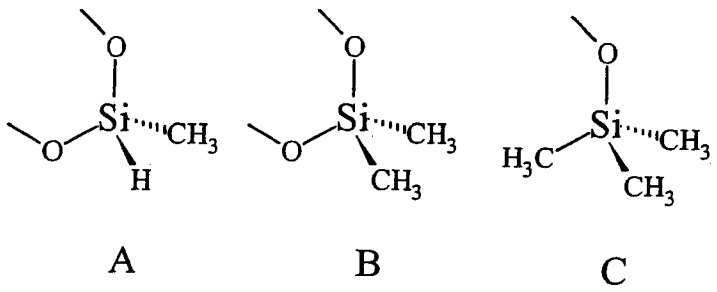

Figure 4. Precursor structures.

2. Process optimization. Besides precursor structure optimization, process conditions were optimized to improve the incorporation of $-CH_3$ group and to enhance the densification of the as-deposited film.

   One method to improve the incorporation of $-CH_3$ group in the film is to apply lower deposition temperature. Figure 5 illustrates how the film stress varies with the deposition temperature. Near k=3.0, the 400°C process yielded a stress around 45 MPa. A deposition temperature decreases, the film stress decreases rapidly. At deposition temperature of 300°C,

the film stress is around 33 MPa with k value of 2.87, significantly lower than the stress at 400°C.

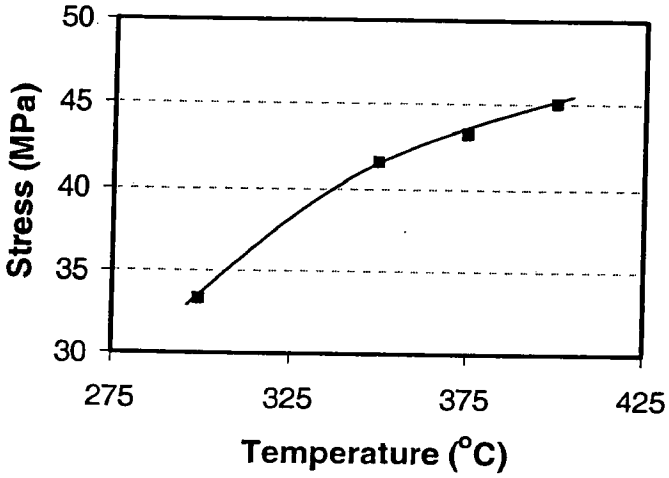

Figure 5. Stress is plotted as a function of deposition temperature of the as deposited films.

The other method to densify the film is to increase the ion bombardment during the PECVD deposition. The increased ion bombardment will not only improve –CH3 incorporation but also enhance the bonding structure of the as-deposited film.

The increased ion bombardment can be achieved by increasing low Frequency (LF) RF power in a dual frequency process. As shown in Figure 6, the film stress decreases linearly as LFRF power percentage in total RF power increases. A negative stress i.e. compressive stress can be achieved at a high percentage of LFRF power.

Haiying Fu Signature

Qingguo Wu Signature

Dong Niu Signature

Inventor Signature

Signature of 1st Witness / Date

Signature of 2nd Witness / Date

Title of Invention: **Methods for producing Low-k OSG Film of low tensile stress**

Date this form was completed:

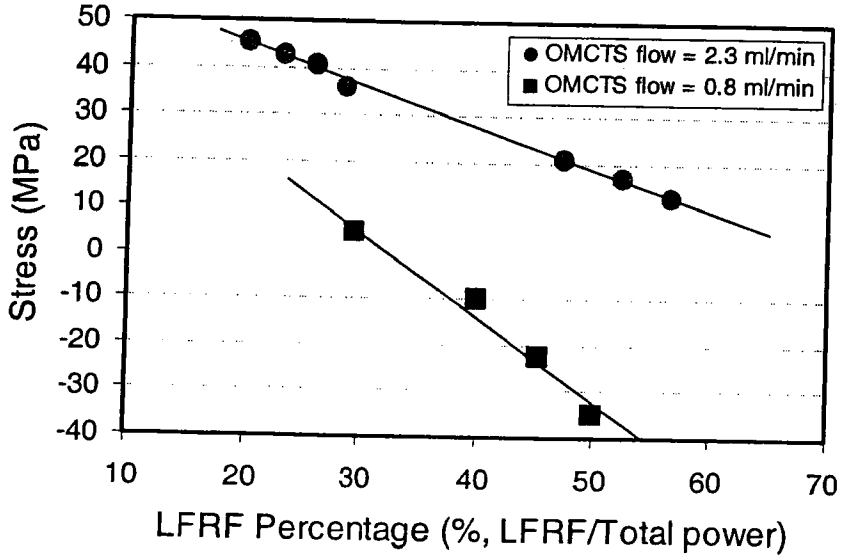

Figure 6. Stress is plotted as a function of LF power of the as deposited films for different precursor flow of OMCTS =2.3ml/min (circles) and OMCTS=0.8ml/min(squares).

Other methods to increase ion bombardment during film deposition include: a) lowering deposition pressure; b) varying showerhead gapping; c) applying pulsed HFRF power, i.e. a HFRF power pulsed at a certain duty cycle.

Haiying Fu Signature

Qingguo Wu Signature

Dong Niu Signature

Inventor Signature

Signature of 1st Witness / Date

Signature of 2nd Witness / Date

INVENTION DISCLOSURE

Novellus Systems, Inc.

Title of Invention: **Methods for producing Low-k OSG Film of low tensile stress**

Date this form was completed:

A low tensile stress of a CDO film will significantly lowers the total tensile stress of the integrated stack, reduces the failure rate due to the high tensile stress, and improves the production yield.

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Qingguo Wu Signature

Dong Niu Signature

Inventor Signature

Signature of 1st Witness / Date

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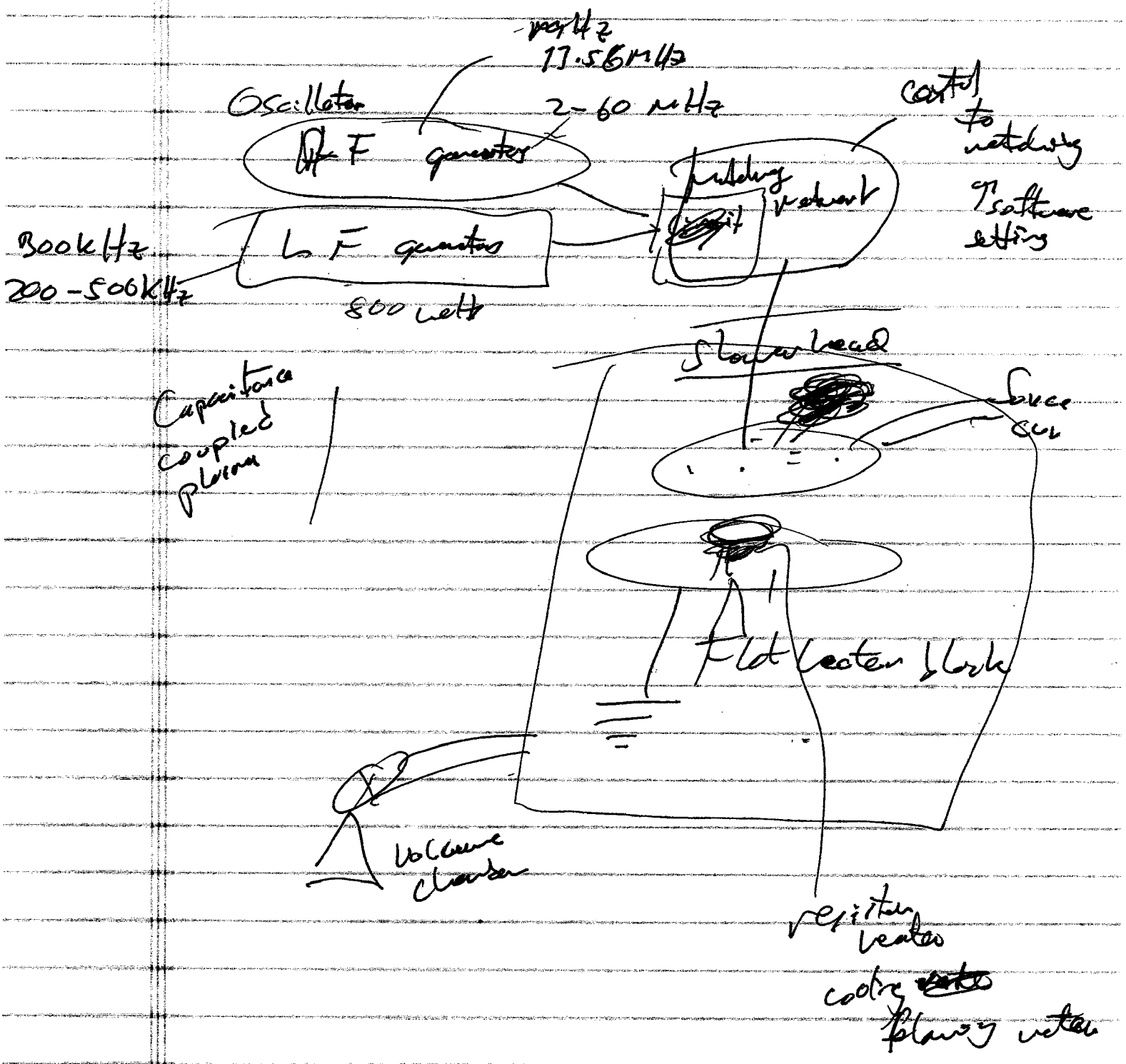
EXHIBIT B

PECVD

flow action & energy action



Increase mean path of ion

lower pressure

lower gapping (velocity)

pulsed duty cycle (gives a ~~de~~ low frequency)

electrically increases ion temp

1 KHz – up 10 KHz

500 Hz

20-80% duty cycle

Pressure – 2 – 20 Torr

2 Torr – 6 Torr preferred

Gap – Will get Hall Effect

EXHIBIT C

BEYER WEAVER & THOMAS, LLP

INTELLECTUAL PROPERTY LAW

590 W. El Camino Real, Mountain View, CA 94040
Telephone: (650) 961-8300 Facsimile: (650) 961-8301
www.beyerlaw.com

FACSIMILE COVER SHEET

Date: 1/12/04

Receiver: JEFF

TEL #:

FAX #: BWT BK

Sender: JOE B.

Re: NOULP094/P091

Pages Including Cover Sheet(s): 2

MESSAGE:

FIG. 13 FOR REVIEW

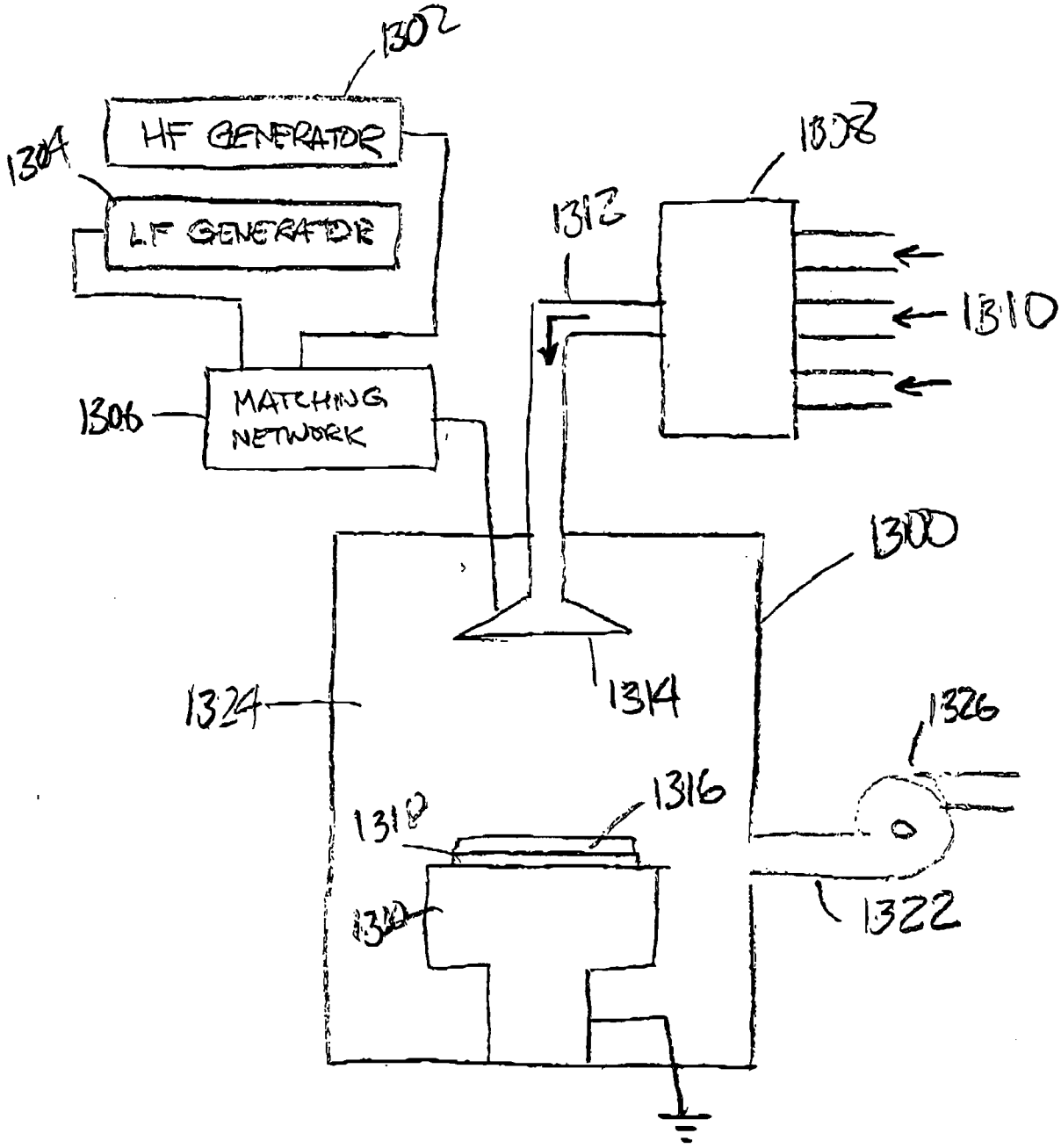

1302
HF GENERATOR
1304
LF GENERATOR
1308
1312
1310
1306
MATCHING NETWORK
1300
1314
1324
1326
1316
1318
1320
1322

FIG. 13

EXHIBIT D

From: Joe Bond
To: Haiying.Fu@NOVELLUS.com
Date: 2/5/2004 4:01:24 PM
Subject: NVLS-2889/NOVLP091 First draft

Haiying FU
Novellus Systems, Inc.
11155 Southwest Leveton Road
Tualatin, OR 97062

Re: Patent Application Entitled: METHODS FOR PRODUCING LOW-K CDO FILMS WITH LOW TENSILE STRESS
Your File: NVLS-2889 Our File: NOVLP091

Dear Haiying:

Enclosed for your review is a draft of the above-identified patent application together with informal versions of our proposed drawings (sent separately today by FAX). The file is password protected using a password that I left on your voicemail at your work number on 5 February, 2004. The drawings are in Microsoft Visio format, which you may have trouble reading. I can convert them to another format if necessary. Alternately, a free visio viewer (vviewer.exe) is available on microsoft's web site.

The draft includes several blanks, which I trust are relatively self-explanatory so that you can readily fill in the required information. Please review the application to ensure that:

(1) it contains an accurate and complete written description of the invention;

(2) it sets forth sufficient detail to enable one skilled in the art to which it pertains to make and use the invention; and
(3) it discloses the best known mode of practicing the invention (i.e., the preferred way of making and using the invention).

To the extent possible, please make any changes to the application as you would like them to appear in the final version. When you have finished reviewing the application, please return the marked-up version of the draft. After we have received your comments and made any appropriate revisions, we will prepare the final version which will be forwarded for your review, together with the formal papers that you will need to execute.

Please note that by law this application (as filed) will be published in the U.S. at 18 months from the earliest priority date. If the application will not be filed internationally, you may choose to request nonpublication, but this request must be made upon filing the application. You also have options of early publication and republication, but as these issues are complex, please telephone us should you have any questions on any aspect of publication.

We have a duty to disclose the most pertinent prior art of which you are aware to the Patent and Trademark Office. If you can think of any pertinent references or patents, or any similar existing technology, please let us know. The duty to disclose prior art continues until the patent actually issues; if you become aware of other prior art in the future, please let us know.

Very truly yours,
BEYER WEAVER & THOMAS, LLP

Joesph E. Bond

Joe Bond

Beyer Weaver & Thomas, LLP
590 W. El Camino Real
Mountain View, CA 94040
(650) 961-8300
JBond@beyerlaw.com

==========

CC: Jeffrey Weaver

EXHIBIT E

From: Jeffrey Weaver
To: Bond, Joe; Fu, Haiying
Subject: Re: Revision of the 2nd patent application

Haiying,

We have gone through your comments on the draft application. It is nearly finalized. Before we send the next draft, I would like to make a few comments and ask a few questions. Possibly we can discuss this late Monday afternoon or Tuesday.

Also - a minor point - at some places we characterize power in Watts and in other places in Watts/cm2. We should be consistent. I'm not sure which cm2 you are referring to. Is it the wafer surface area?

Hopefully, we can wrap this up early next week.

Jeff

>>> "Fu, Haiying" <Haiying.Fu@NOVELLUS.com> 2/22/2004 6:53:33 PM >>>
****
Warning: Your file, NOVLP091a.app.zip/NOVLP091a.app.doc, is password-protected. It was not scanned by InterScan.
**********_**********

<<NOVLP091a.app.zip>>
Joe and Jeff:

I did some modification on the 2nd patent application. Please review and modify based on the previous one. This one will be emphasizing on deposition method while continuing to address preferred precursor choice.

The password is the same as the one you gave me.

Thanks,
Haiying

EXHIBIT F

BEYER WEAVER & THOMAS, LLP

INTELLECTUAL PROPERTY LAW

590 W. El Camino Real, Mountain View, CA 94040
Telephone: (650) 961-8300 Facsimile: (650) 961-8301
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March 29, 2004

Via Federal Express

Haiying Fu
Novellus Systems, Inc.
11155 Southwest Leveton Road
Tualatin, OR 97062

Re: U.S. Patent Application Entitled: METHODS FOR PRODUCING LOW-K CDO FILMS WITH LOW RESIDUAL STRESS
Your File: NVLS-002889
Our File: NOVLP091

Dear Haiying:

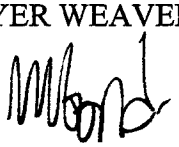
Thank you for your comments concerning the above-referenced patent application. We have now revised the application in accordance with your comments.

At this time it is necessary to have each inventor sign the enclosed standard forms. One form is a Declaration and Power of Attorney form, and the other form is an Assignment of rights to Novellus Systems, Inc. After the final read through of the patent application (assuming no additional changes are needed), please have each inventor read and then sign and date each of the enclosed forms by their name. **Afterwards, please return the application together with the executed documents so that we may file them with the U.S. Patent and Trademark Office.**

Finally, we would again like to remind you of our duty to disclose the most pertinent prior art of which you are aware to the Patent and Trademark Office. If you can think of any pertinent references or patents, or any similar existing technology, please let us know. The duty to disclose prior art continues until the patent actually issues; if you become aware of other prior art in the future, please let us know.

Best regards,

BEYER WEAVER & THOMAS, LLP



Joseph E. Bond

JEB/lmd
Enclosures